Structure-Sensitive Properties of Advanced Permanent Magnet Materials: Experiment and Theory

DESCRIPTION: This program, a joint endeavor between BNL and INEEL, elucidates the relationships linking the micro-structure of advanced permanent magnetic materials to their magnetic properties, with the ultimate goal of understanding how to synthesize desirable microstructures by suitable processing methods. The scope of the research has expanded to include studies of related magnetic alloys in their nanocrystalline, nanocomposite and amorphous forms. This expanded scope will form the core of the magnetic nanomaterials research in the proposed BNL Nanoscience Center. Extensive collaboration with other DOE laboratories, academia and industry is a strong component of the research.

SELECTED PROGRAM HIGHLIGHTS:

- ***Effects of Intergranular Interactions in Nanocrystalline Permanent Magnets:*** Experimental and computational results indicate that formation of multi-grain "interaction domains" in nanoscale magnetic materials significantly alters the nature of the magnetic reversal. (D. C. Crew, L. H. Lewis and V. Panchanathan, "Multi-scale magnetic domains observed in die-upset melt-spun magnets using magnetic force microcopy" *J. Magn. Magn. Mater*. **231** (1) (2001) 57).
- ***Manipulation of Interphase Exchange in Magnetic Nanocomposites***: Results concerning magnetic reversal in $L1_0$-type CoPt/Co thin film bilayers indicate the ability to tailor the response of the magnetic "exchange-spring". (D. C. Crew, J. Kim, L. H. Lewis and K. Barmak, "Interdiffusion in Bilayer CoPt/Co Films: Potential for Tailoring the Magnetic Exchange Spring", *J. Magn. Magn. Mater*. **233** (2001) 257-273).
- ***Processing-Induced Internal Stress in Magnetic Nanocomposites:*** Quantitative analysis of synchrotron XRD data from rapidly-solidified $Nd_2Fe_{14}B$-based nanocomposites indicates significant strain that affects the magnetic response. (L. H. Lewis, A. R. Moodenbaugh, D. O. Welch & V. Panchanathan, "Stress, Strain and Technical Magnetic Properties in "Exchange-Spring" $Nd_2Fe_{14}B + \alpha$-Fe Nanocomposite Magnets", *J. Phys. D: Appl. Phys*. **34** (2001) 744-751).

IMPACT: *Challenged long-standing assumptons about structure-property relationships in permanent magnets:*

- Championed the (unpopular) idea that high coercivity and high remanence are mutually incompatible in nanoscale magnets by virtue of intergranular exchange interactions.
- Promoted the idea that "amorphous" materials are usually not truly amorphous and often, if not always, contain angstrom-scale nuclei that are extremely challenging to detect. These quenched-in nuclei ultimately determine both the as-processed magnetic properties as well as the devitrification pathways.
- 2 patent disclosures: The "Double" Magnetocaloric Effect; Novel Hysteresis Mechanism in Hybrid Permanent Magnets

INTERACTIONS:

- *BNL:* Y. Zhu (Materials Sci. Dept.): advanced electron microscopy; J. Hill (Physics Dept.) and C. C. Kao (NSLS): magnetic nanodispersions.
- *DOE National Lab Collaborations*: Core Participant in **DOE Centers for Excellence in Synthesis and Processing**: "Tailored Microstructures in Hard Magnets"; "Isolated & Collective Phenomena in Nanocomposite Magnets."; **Ames Lab** (R. W. McCallum, M. J. Kramer: solidification studies in melt-spun NdFeB & ferro. bulk amorphous glasses);
- *U. S. Academic Collaborations:* **Carnegie Mellon University** (K. Barmak: reversal studies in model bilayer systems; S. Majetich: nanoparticles; M. McHenry: general student exchange), **SUNY Stony Brook** (R. J. Gambino: magnetic nanocomposite films, S. Sampath: thermal spray products; J. Parise: nano-oxides);
- *Industrial Collaborations:* **Magnequench International, Inc**. (V. Panchanathan, B. M. Ma: general structure/property interrelationships in $Nd_2Fe_{14}B$-based nanocrystalline magnets);
- *Educational Activities*: SUNY SB "Seed Grants" (2); DOE Community College Institute: 2 students (1999); DOE Energy Research Undergraduate Laboratory Fellowship (ERULF) Program: 3 students since 1999; co-supervised 2 Ph.D. students (Lehigh University, SUNY Stony Brook); NSF Division of International Programs/East Asia.

PERSONNEL:
L. H. Lewis (BNL, experimental studies), **D. O. Welch** (BNL, theoretical studies), **D. Crew** (BNL, post-doctoral associate, 1999-2000), **C. Harland** (BNL, post-doctoral associate, 2001-); **D. J. Branagan** (INEEL, experimental studies), **B. Meacham** (INEEL, post-doctoral associate, 2001-); also **J. van Lierop** (BNL LDRD, "Magnetic Nanodispersions") and **M.-H. Yu** (BNL Royalty income project, "Magnetocaloric Effect in Nanocomposites")

RECOGNITION

- 8 invited presentations (BNL).
- Symp. organizer, 1999 Spring MRS Meeting: "Hard Magnets: Principles, Materials, Processing" (Lewis)
- Program Committees, MMM/Intermag conferences:1999- 2001. (Lewis)
- Inter'l Advisory Committee, Inter'l Training Inst. for Mat. Sci., Vietnam.
- TMS Young Leader Award 2000 in the Electronic, Magnetic, and Photonic Materials Division (D. J. Branagan)

BUDGET: Core BES Program: (FY 2002) $410 K total; $333 K BNL and $77 K INEEL.

Structure-Sensitive Properties of Advanced Permanent Magnet Materials: Experiment and Theory

Scientific Staff, Areas of Expertise:

BNL:

Laura H. Lewis	Basic and functional properties of magnetic properties of materials; rapidly solidified materials, solid state chemistry and structure of pnictides, oxides and chalcogenides.
David O. Welch	Theoretical materials science; crystal lattice defects; superconducting and magnetic materials; statistical thermodynamics and kinetics.

INEEL:

Daniel J. Branagan	Experimental aspects of rapid solidification processing and metallic glass formation; design of advanced multicomponent alloys for selected applications; nanoscale science and technology, general materials characterization.

Number of support staff:

Post-doctoral researchers: 2 (1 at BNL, 1 at INEEL)
Technical support staff: 1.25 (0.25 at BNL, 1 at INEEL)

Other financial support:

NSF Division of International Programs/East Asia (invited): $75 K (1998 – 2001); BNL LDRD FY 2002: $73 K; BNL Patent Royalty Income FY 2001 – 2002: $125 K.

Future Research Directions: The research directions outlined below will naturally segue into the core research on magnetic nanomaterials in the proposed DOE Nanoscience Center.

- Expand present research on bulk magnetic materials to include investigations of coercivity mechanisms in multi-phase nanocaled or largely amorphous alloys comprised of ferromagnetic and ferri- or antiferromagnetic phases.

- Continue with newly-initiated program on “Magnetic Nanodispersion” thin film systems of ferromagnetic and antiferromagnetic oxide phases to elucidate factors controlling the exchange and magnetostatic interactions inherent to these systems. Once established, the work will be extended to include thin film systems of a highly-polarizable, but not ferromagnetic, matrix with antiferromagnetic oxide phases.

- Commence with already-planned collaborative investigations of magnetic behavior of nanodisperse Co, Fe, CoPt and FePt nanoparticles synthesized by the polyol method at Carnegie Mellon University. Properties to be initially investigated include uniformity of particle size, interparticulate interactions and the properties of the blocking temperature.

- Connect *ac* susceptibility measurements to details of the micro- or nanostructure in overquenched (*i.e.*, amorphous/ nanocrystalline) Nd-Fe-B rapidly-solidified materials.

- Apply experience and results gained from work on permanent magnetic nanocomposites to magnetic nanomaterials with novel or multiple functionalities, such as enhanced magnetocaloric properties or simultaneous ferromagnetic and ferroelectric properties (*i.e.*, multiferroics). Plans include further investigation of “core-shell” magnetic nanocomposite particle structures of CrO_2+Y-Al-garnet oxides.

Research Facilities & Equipment:

Quantum Design MPMS 5-T SQUID Magnetometer with *ac* and high moment options and furnace insert; Walker Scientific Hysteresisgraph; TA Instruments Thermal Analysis (DSC + DTA/TGA); Digital Instruments Nanoscope III Tapping Mode Atomic/Magnetic Force Microscope; Heat Capacity measurement option for Quantum Design PPMS system.

Structure-Sensitive Properties of Advanced Permanent Magnet Materials:
Experiment and Theory

List of Collaborators and Institutions:

Brookhaven National Laboratory:	
Yimei Zhu (Materials Science Dept.):	High-resolution electron microscopy, Lorentz imaging
John Hill (Physics Department), Chi-Chang Kao (NSLS):	Synchrotron-based characterization of magnetic thin-film nanodispersions and magnetic nanoparticles.
DOE National Laboratories:	
R. William McCallum & Matthew J. Kramer, Ames Laboratory	Fundamental studies of rapid solid ification of $Nd_2Fe_{14}B$-based materials, technical and fundamental properties of ferromagnetic bulk amorphous glasses, exchange-bias phenomena in bulk multiphase alloys.
Academia:	
R. J. Gambino, SUNY Stony Brook M. Aronson, Univ. Michigan	Magnetic interactions in thin-film magnetic nanodispersions of Ni/NiO, Co/MnO and Pd/MnO.
S. Sampath, J. Parise SUNY Stony Brook	Functional magnetic nanocomposite oxides
Katayun Barmak, Carnegie Mellon University	Magnetic reversal and crystallographic attributes of CoPt composite thin films and CoPt/Co model bilayers.
Sara Majetich Carnegie Mellon University	Interactions in Co, Fe and CoPt, FePt nano-particles synthesized by the polyol method.
Michael McHenry Carnegie Mellon University	Student exchange, fundamental materials science of magnetic materials.
Industry:	
Bao-Min Ma, Prem Panchanathan (fm) Magnequench International, Inc.	General structure/property interrelationships in melt-quenched $Nd_2Fe_{14}B$-based nanocrystalline materials.

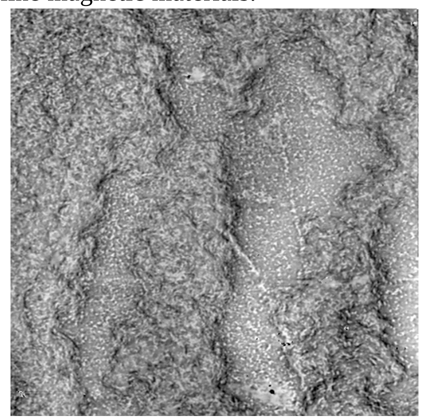
Structure-Sensitive Properties of Advanced Permanent Magnet Materials: Experiment and Theory

Research Highlights:

- ***Effects of Intergranular Interactions in Nanocrystalline Permanent Magnets***

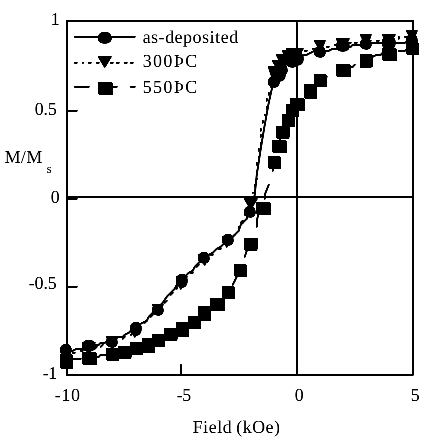
The energy product $(BH)_{max}$ quantifies the maximum amount of useful work that can be performed by a magnet. The energy product is determined by the magnetic remanence and the coercivity which, as extrinsic properties, are derived from the magnets' microstructure. Thus, in principle, magnetic material microstructures may be tailored to obtain defined parameters to produce optimal permanent magnets. However, as asserted by the eponymous Murphy, "Nature favors the hidden flaw": evidence acquired through experimental and computational investigation strongly suggests that maximum remanence and maximum coercivity are mutually exclusive in nanocrystalline magnetic materials.

Investigations performed on $Nd_2Fe_{14}B$-based nanocrystalline materials that were thermomechanically-deformed to produce systematically-varying levels of relative particle alignment reveal an increase in the size of the multi-grain "interaction" magnetic domains with increasing level of alignment from magnetic force microscopy experiments (D. C. Crew, L. H. Lewis and V. Panchanathan, "Multi-scale magnetic domains observed in die-upset melt-spun magnets using magnetic force microcopy" *J. Magn. Magn. Mater*. **231** (1) (2001) 57) and simple micro-magnetic simulation (D. C. Crew and L. H. Lewis, "Effect of grain alignment on magnetic structure in nanoscale material", *IEEE Trans. Magn*. **37** (4) 2512 (2001)) These investigations conclude that nanocrystallite alignment favors exchange-induced coupling of magnetic moments at the grain edges, producing an increased interaction domain size. However, at the same time the reduction of the perpendicular magnetization component across the intergrain interface tends to reduce the resistance to magnetic reversal and thereby lowers coercivity



Magnetic Force Microscopy (MFM) 50 x 50 µm image of a thermomechanically-deformed nanocrystalline magnet of $Nd_2Fe_{14}B$ in the coercive state. The with easy-axis alignment of the crystallites is perpendicular to the plane of the page. The image shows clusters of exchange-coupled crystallites that group together to form large, elongated "interaction domains" that reverse easily under the application of a reverse field.

- ***Manipulation of Interphase Exchange in Magnetic Nanocomposites***



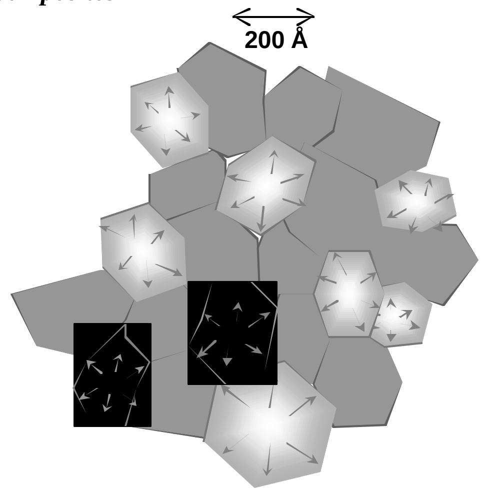
Hysteresis loops for CoPt/Co (25/16.7 nm) bilayer thin films with the Co layer in three different annealing states (as-deposited, 300 and 550°C) that illustrate the progression from two-stage magnetic reversal with single-stage magnetic reversal upon annealing at 550 °C).

Magnetic nanocomposites comprised of a magnetically-hard, low-magnetization phase combined on the nanoscale with a magnetically-soft, high-magnetization phase are anticipated as the next-generation permanent magnet. The performance of this class of magnets is necessarily determined by the detailed nature of the interphase interface, which is extremely difficult to study in bulk form, but may be accessed in thin-film systems. Significant improvement of the interlayer exchange was produced by moderate annealing of the bilayer thin films, To this end, thin-film layers of crystallographically-ordered, very high anisotropy $L1_0$-type CoPt of varying thicknesses were capped with low-anisotropy Co to produce model thin-film bilayers in order to study the effects of microstructural parameters on magnetic exchange and reversalas revealed by magnetic force microscopy, transmission electron and bulk magnetic measurement. It was revealed that annealing-induced interdiffusion altered the interphase interface which altered the system anisotropy and magnetic behavior.

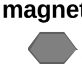
To support these conclusions micromagnetic modeling of different bilayer conditions was performed that indeed confirmed interfacial modification can enhance interlayer exchange. (D. C. Crew, J. Kim, L. H. Lewis and K. Barmak, "Interdiffusion in Bilayer CoPt/Co Films: Potential for Tailoring the Magnetic Exchange Spring", *J. Magn. Magn. Mater*. **233** (2001) 257-273.) Results of this study revealed the potential of careful interphase interfacial control for optimizing the exchange-spring magnet.

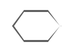
- ***Processing-Induced Internal Stress in Magnetic Nanocomposites***

In addition to their technological attributes, magnetic nanocomposite materials exhibit interesting, non-intuitive physical properties such as an apparent increased Curie temperature of the lower-T_C constituent and anomalous high-temperature coercivities. To investigate the origin of these phenomena quantification of the internal strain state of the individual phases in melt-quenched $Nd_2Fe_{14}B + \alpha$-Fe nanocomposites was undertaken. Synchrotron x-ray diffraction studies were performed at the NSLS, and the data was analyzed within the framework of a modified Williamson-Hall method. It was determined that the constituent $Nd_2Fe_{14}B$ grains are largely strain-free, but the α-Fe component exhibited significant stress (~1 GPa) with an associated strain of approximately 0.1%. The results of this study helped to confirm that the enhanced T_C of $Nd_2Fe_{14}B$ was likely an exchange- and not stress-induced effect as well as provide a basis for the marginal remanence enhancement and anomalous elevated-temperature coercivities found in these nanocomposites. (L. H. Lewis, A. R. Moodenbaugh, D. O. Welch and V. Panchanathan, "Stress, Strain and Technical Magnetic Properties in "Exchange-Spring" $Nd_2Fe_{14}B + \alpha$-Fe Nanocomposite Magnets", *J. Phys. D: Appl. Phys*. **34** (2001) 744-751.)



Schematic view of melt-quenched permanent magnetic nanocomposite.





Arrows indicate local tensile forces

“Structure-Sensitive Properties of Advanced Permanent Magnet Materials: Experiment and Theory”
Publication List: 1999 – present

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2. J. van Lierop, L. H. Lewis, K. E. Williams and R. J. Gambino, “Magnetic exchange effects in a nanocomposite Ni/NiO film”, *J. Appl. Phys*., in press.

3. V. V. Volkov, D.C.Crew, Y. Zhu and L. H. Lewis, “Magnetic field calibration of a transmission electron microscope using a permanent magnetic material”; *Rev. Sci. Instrum*., accepted.

4. M. J. Kramer, L. H. Lewis, L. M. Fabiette, Y. Tang, K. W. Dennis and R. W. McCallum, “Solidification and its role on the mechanisms of microstructural refinement and magnetism in $Nd_2Fe_{14}B$”; *J. Magn. Magn. Mater*., submitted.

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6. Laura H. Lewis and David C. Crew, "The Coercivity – Remanence Tradeoff in Nanocrystalline Permanent Magnets", invited paper, to appear in MRS Spring 2001 Proceedings.

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19. D. J. Branagan, M. J. Kramer, Yali Tang, R. W. McCallum, D. C. Crew and L. H. Lewis, "Engineering Magnetic Nanocomposite Microstructures", *J. Materials Science*, 35(14): 3459-3466, July 2000.

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